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Major Goals: The major goal of this DURIP was the development of a universal optical chamber for studying a variety of combustion problems from sub-atmospheric to high pressures (50,000 psi) that can provide burning rate data, visual observations, a platform for evaluating diagnostics, and additional data for model development.

Accomplishments: The major accomplishments of this DURIP were the design and fabrication of a combustion chamber with optical access, hydrostatically pressure tested to 55,000 psi, the accompanying gas flow and control systems, and an insert module for studying solid and liquid propellant combustion.

Training Opportunities: Nothing to Report

Results Dissemination: "Coflow and Counterflow Burning Rate Measurements of Energetic Materials at Elevated Pressures," Richard A. Yetter, Grant A. Risha, J. Eric Boyer, and Gregory Derk, 48th Combustion / 46th Airbreathing Propulsion / 36th Exhaust Plume and Signatures / 30th Propulsion Systems Hazards Joint Subcommittee Meeting, and Programmatic and Industrial Base Meeting, JANNAF, 4 – 7 December 2017, Newport News Marriott at City Center in Newport News, Virginia.

Honors and Awards: Richard Yetter was elected Fellow of the ASME and the Combustion Institute

Protocol Activity Status:

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14. ABSTRACT
A new experimental chamber has been developed for studying combustion phenomena of advanced energetic materials. This Ultra-High Pressure Optical Chamber (UHPOC) has an operational pressure from 0.03 to 345 MPa and is optically-accessible to facilitate visual observation of the combustion process. Experimentally, the linear burning rate of propellants and explosives is measured directly using high-speed cinematography. The UHPOC has a working volume of 0.012 m^3 (12 liters) allowing for a variety of experimental configurations and constant pressure conditions. The chamber's modularity allows a single apparatus to study solid propellant strand combustion with coflow and/or counterflow configurations, liquid propellant strand combustion, and homogeneous gas combustion. This unique experimental facility also provides a platform for developing diagnostics for use at ultra-high pressures.

15. SUBJECT TERMS
High Pressure, Energetic Materials, Propellants, Burning Rate

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Introduction

The dependence of burning rate on pressure is of fundamental importance to ballistic properties. The linear burning rate of a propellant or explosive is often described over a specific pressure range by the empirical equation $r_b = aP^n$. The parameter a is often considered a function of temperature, while the exponent n is independent of temperature and describes the influence of pressure on the burning rate. Burning rate data of propellants and explosives have been measured for years using various experimental techniques. The photocinemicrographic and closed bomb combustion methods are two common approaches for measuring the burning rate. In the photocinemicrographic method, the energetic material sample, usually in the form of a strand, is pressurized in the combustion vessel to a desired pressure, and a video of the sample regression is recorded for analysis of the burning rate. A small sample is used such that the volume of gas produced from combustion does not contribute significantly to the overall volume of the chamber, and thus, the sample is assumed to burn at nearly constant pressure. The upper limit for these windowed chamber experiments has generally been about 50 MPa, although most of the limited data available are for 10 MPa and lower. For pressures higher than 50 MPa, the closed bomb technique is used in which the sample, in the form of a powder or strand, burns in a relatively small volume without observation. Pressure-time data are used to deduce the burning rate from a model developed to describe the experiment that must account for variable thermochemistry, heat loss, ignition, and flame spread.

Examples of the burning rate dependence on pressure of several monopropellants are shown in Figures 1-4. Figure 1 shows the burning rates of octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) and 1,3,5-trinitroperhydro-1,3,5-triazine (RDX) as a function of pressure, where it is observed that the empirical burning rate equation can nearly correlate the data with a single pressure exponent. Between 10 and 70 MPa, some variation in the value of n may exist as three different experiments were required to achieve the entire pressure range for HMX.

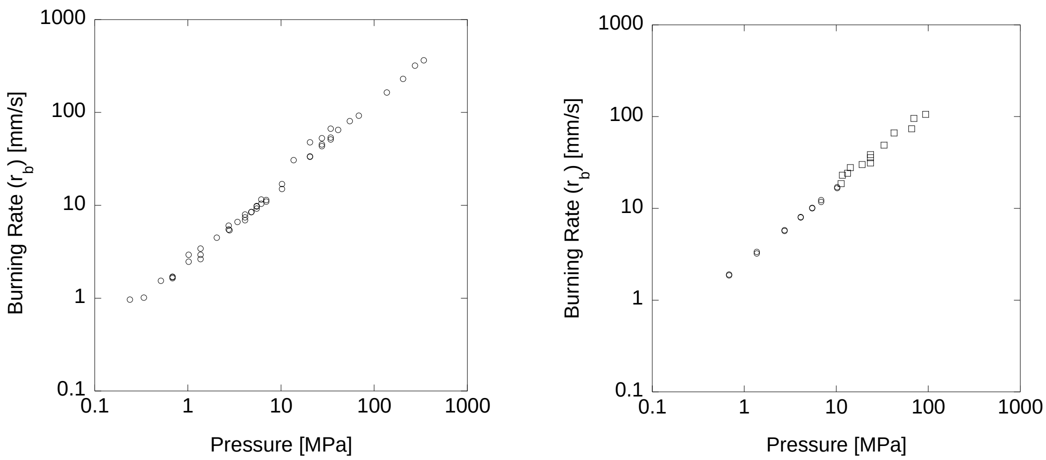

Figure 1. (left) HMX burning rate at ambient temperature;
(right) RDX burning rate at ambient temperature [1].

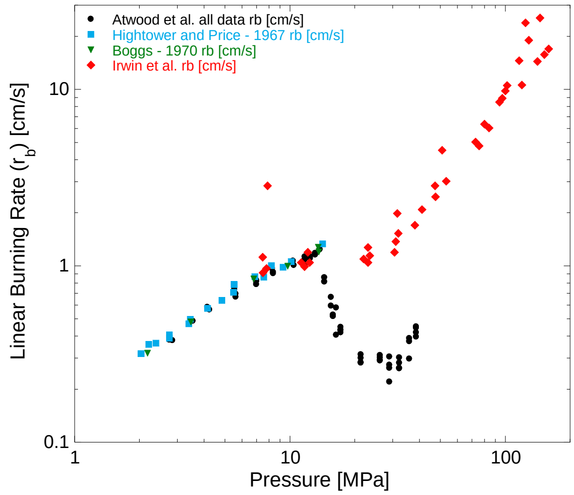

Figure 2. AP burning rate at ambient temperature [1]. References for the Hightower and Price, Boggs, and Irwin et al. data may be attained from Atwood et al. [1].

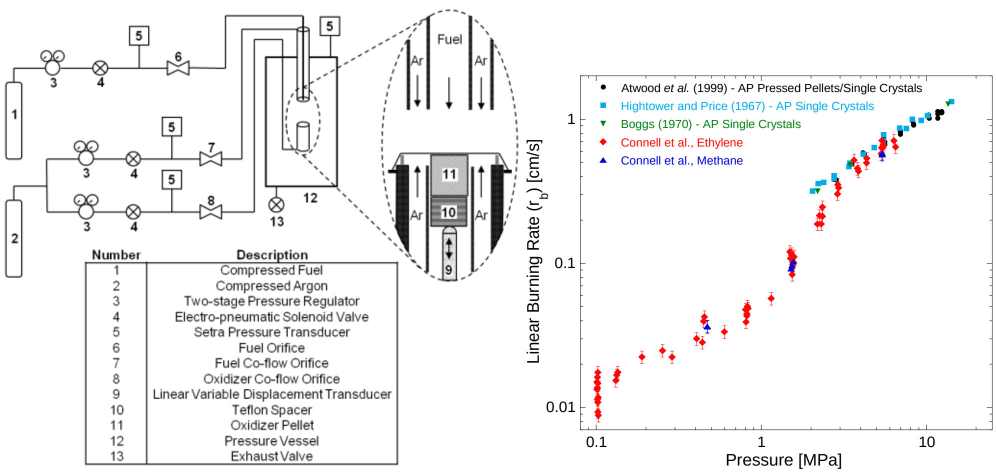

Figure 3. (left) Experimental facility for pressurized counterflow strand burning measurements. (right) AP burning rate below the low pressure self-deflagration limit supported by counterflow diffusion flame. Note, at pressures above the self-deflagration limit, the AP burning rate reestablishes the values of Figure 2 [2]. References for the Atwood et al., Hightower and Price, and Boggs data for burning rates of AP pellets may be attained from [1].

Figure 2 shows regression rate data for ammonium perchlorate (AP) at ambient temperature where the low pressure deflagration limit is shown to be approximately 2 MPa. The negative slope of the curve between 10 and 30 MPa has been explained by unstable combustion and changes in the chemical mechanism. Above 30 MPa, the burning rate again increases with pressure, having a greater pressure exponent than the pressure dependence below 10 MPa.

The pressure dependence below the self-deflagration limit can be achieved from counterflow burning rate experiments as illustrated in Figure 3, in which hydrocarbon gases are counterflowed against the decomposition gases from the AP monopropellant flame to produce a stable diffusion flame that supports the monopropellant flame. As seen in the figure, between 0.1 and 1 MPa, the pressure exponent is nearly the same as above the self-deflagration limit from 2 to 10 MPa, while the pre-exponential factor *a* is about an order of magnitude lower. In between 1 and 2 MPa, Figure 3 shows a high pressure exponent. It is interesting to note that the AP burning rate in the counterflow experiment essentially represents the burning rate of an infinite diameter particle in an AP based composite propellant.

Figure 4 presents burning rate data for liquid nitromethane from atmospheric pressure to approximately 200 MPa. Only the data from atmospheric pressure to 12 MPa was attained with an optical chamber. The data show two slope breaks: one at 15 MPa and the other at 70 MPa. While the kinetic mechanisms of nitromethane are considered relatively well known, current models do not predict this behavior. One consideration is the lower pressure burning rate is conventional burning with an interface between the liquid and gas, while the higher pressure burning rate occurs without an interface, that is, under supercritical conditions, and the higher slope in between represents the transition region.

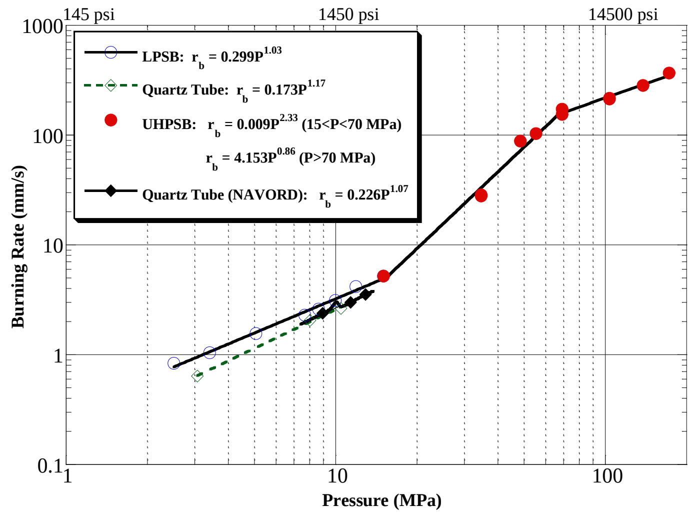

Figure 4. Liquid nitromethane burning rates at ambient temperature [3].

Figure 5 shows the burning rates of liquid hydroxyl ammonium nitrate (HAN)-water solutions and HAN/H_2O/CH_3OH mixtures as a function of pressure. As evident from Figure 5, the burning rate pressure dependence is strongly affected by the water content and the addition of a fuel to the mixture, again emphasizing significant changes in combustion behavior with pressure.

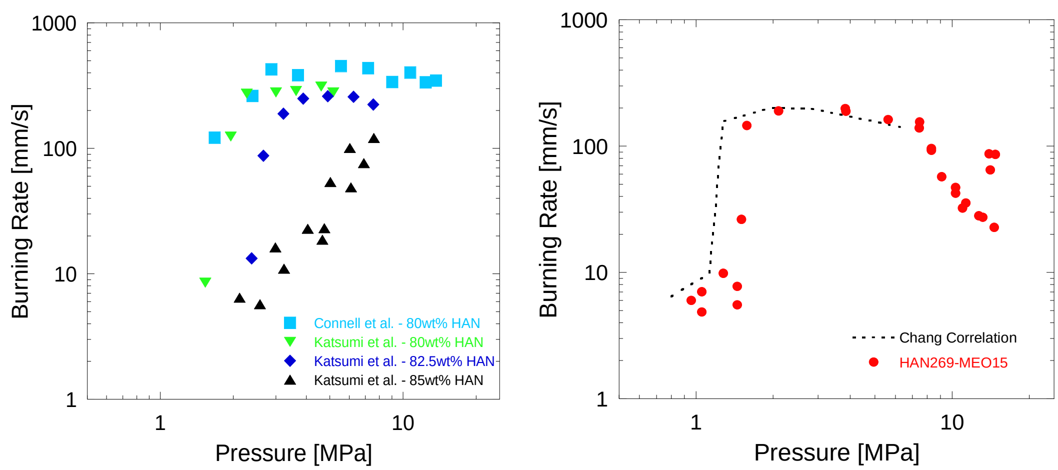

Figure 5. (left) HAN/water burning rates at ambient temperature with various weight percentages of HAN; (right) HAN/H2O/CH3OH burning rate at ambient temperature [4,5].

These limited examples illustrate the impact pressure has on burning rate. As can be seen from much of the data, the empirical equation $r_b = aP^n$ is valid for only limited pressure ranges. Composite propellants are also well known to exhibit slope breaks, burning rate plateaus, and even negative pressure dependencies. Most burning rate data are limited to pressures below 20 MPa, *and for data greater than 50 MPa, observations of the burning processes are not available*, and thus the presence of non-ideal burning (e.g., burning in cracks, side burning, etc.) can only be surmised. In addition, the manner in which burning rate changes with pressure when the temperature of the energetic material is varied (burning rate temperature sensitivity) can also change the dependence of burning rate on pressure. Understanding of burning rate pressure and temperature sensitivity are key elements to propellant combustion response and stability as evident from a Zeldovich-Novozhilov analysis [6].

The ability to predict the linear burning rate of an energetic material as a function of pressure and initial temperature is a highly desirable goal. Such predictions depend on understandings of thermochemical equations of state, condensed phase chemistry, gas-phase chemistry, and the interfacial chemistry that exists between phases. It is important to note that none of the unique pressure dependencies of the above examples have been predicted by detailed combustion models. Furthermore, it is critical to the body of knowledge regarding energetic materials to promote significant developments of next generation technologies that provide advancement of the synthesis of new materials and propellants and the development of various models to predict ballistic behavior for various defense systems. These advancements can extend/impact current capabilities and provide next generation concepts and developments for future systems such as missile, gun, and rocket technologies.

Program Objective

The objective of this DURIP was to develop a new facility for the study of energetic material combustion over a pressure range of 0.03 MPa to 300 MPa that allows for direct observation of the combustion process and determination of ballistic parameters. The experimental facility was based upon extensive experience with university laboratory scale high-pressure combustion chambers [7-11] and the design and manufacturing experience of HiP. The high-pressure optical combustion chamber will be used to study the ignition and combustion phenomena associated with advanced energetic materials. Being optically accessible, important combustion phenomena can be observed and the burning rate can be directly measured using high-speed cinematography. The strand burner has many desirable experimental capabilities, such as broad pressure, controlled purge flow rates, and types of purge gas.

Beyond solid propellant burning rate data, this optical pressure vessel offers other desirable diagnostic opportunities. For example, detailed surface observations can be made using a propellant feed system to maintain the propellant surface at one location. Observations such as close-up photographs of the flame structure and/or propellant surface can be ascertained during the combustion event. At low pressures, it is possible to incorporate laser techniques to obtain species concentrations in the product stream, and, ultimately, flame temperatures. High speed cameras and laser or x-ray techniques also allow analysis of the interfacial burning dynamics of composite propellants at high pressures. In addition to conventional strands, the vessel has the capability to operate in an opposed flow type configuration. Such configurations allow studies of the transition in burning behavior across the low pressure deflagration limit, by establishing a diffusion flame off of the burning surface. They can also be used in strain rate studies and in studies on extinction and ignition. The chamber is based on a modular vessel design so that liquids and gases can be studied as well as solids. The working volume of the design enables modular inserts that allow a single facility to study solid propellant strand combustion with coflow and/or counterflow configurations, liquid propellant strand combustion, and homogeneous gas combustion. For this DURIP, only the strand burner insert was developed.

Chamber Design and Manufacture

Design

The Ultra-High Pressure Optical Chamber (UHPOC) was designed for general application in the study of the combustion of energetic materials and/or propellant strands, under constant pressure and room temperature conditions, using multiple methods of ignition including conventional nichrome wire and laser-induced ignition. The chamber design incorporates modularity in order to be utilized for a number of different combustion experiments such as: (1) strand burner experiments that allow burning rate of solids, liquids, and gels to be characterized as a function of pressure and initial temperature. The linear burning rate is measured directly using high-speed cinematography; (2) opposed flow burning to examine the combustion phenomena of opposing reactants at specified separation distances. These can be solid-solid reactants, solid-gel, solid-gas, or gel-gas. Such configurations allow studies of the transition in burning behavior across the low pressure deflagration limit by establishing a diffusion flame off of the burning surface, studies on the effects of strain rates, and studies on extinction and ignition; and (3) premixed flame propagation through constant diameter tubes.

During the design phase of UHPOC system, there were nine specific objectives and requirements:

1. Free volume of no less than 10 liters
2. Maximum working pressure of 345 MPa (50 ksi)
3. Two diametrically opposed optical viewing ports, greater than 1-in diameter, with the ability to accept steel window blanks, acrylic, quartz, or sapphire windows
4. Room temperature operation with varying pressure; no temperature conditioning unit required
5. Fully operational remote operation control console and test stand capable of computer or manual control
6. Key control to arm ignition circuit
7. DAQ acquisition system and pressure monitoring program
8. Rupture disk pressure relief mechanism
9. Modular vessel design so that liquids and gases can be studied as well as solids which allow inserts to study solid propellant strand combustion with co-flow and/or counterflow configurations, liquid propellant strand combustion, and homogeneous gas combustion

In order to successfully operate this optical strand burner, additional equipment was required for its operation. This equipment, discussed in subsequent sections, include: 1) a mounting frame for assembly/disassembly of the chamber section(s) for normal sample loading; 2) a purge gas pressurization / exhaust system, in order to provide the desired pressure environment for studying propellant combustion, and 3) computer / manual remote control of the system.

The UHPOC was designed with many safety aspects in mind since the chamber and windows will experience pressures up to 345 MPa via a gas pressurant; in contrast, existing ultra-high pressure systems use incompressible liquids as the pressurant. The UHPOC is a high-pressure vessel of considerable volume, and consequently has substantial stored energy when pressurized. In addition, the chamber has several interfaces for both the body and the windows, and is connected to external high-pressure plumbing at multiple points. To mitigate the issues associated with multiple interfaces, the chamber has been designed for operation either as a vented pressure vessel or a closed bomb, depending on the nature of the experiment.

Manufacturing

The chamber was manufactured by the High Pressure Equipment Company (HiP). HiP has been a leading manufacturer of ultra-high pressure chambers for the past 65 years. The chamber design and window housings were adopted from one of their most common systems, the R5 series reactors. The design is extremely reliable and robust. According to HIP's documentation, sealing is accomplished by employing a combination of high durometer O-ring and a metal back-up ring. The back-up ring is designed for expansion or contraction depending on the pressure exposure and keeps the O-ring confined with no extrusion clearance. Very low torque is required for a reliable seal, so the sealing mechanisms are very easy to assemble and disassemble. Figure 6 shows a cutaway drawing of the chambers design.

Upon completion of the manufacturing process, raw material (4340), forged stock, and heat treatment certifications and test data were provided from the supply chain by the fabricator (HiP) for the chamber and closure components. The final assembled custom R5 reactor chamber was tested by HiP using a hydrostatic test protocol. Per the test report, this pressure test used water at 55,000 psi and showed no leakage or pressure loss over a 5-minute duration. For the purposes of this proof test, a solid window plug (17-4PH) was used. The system design, materials, and testing resulted in the manufacturer giving a chamber pressure rating of 50,000 psi at 100 °F.

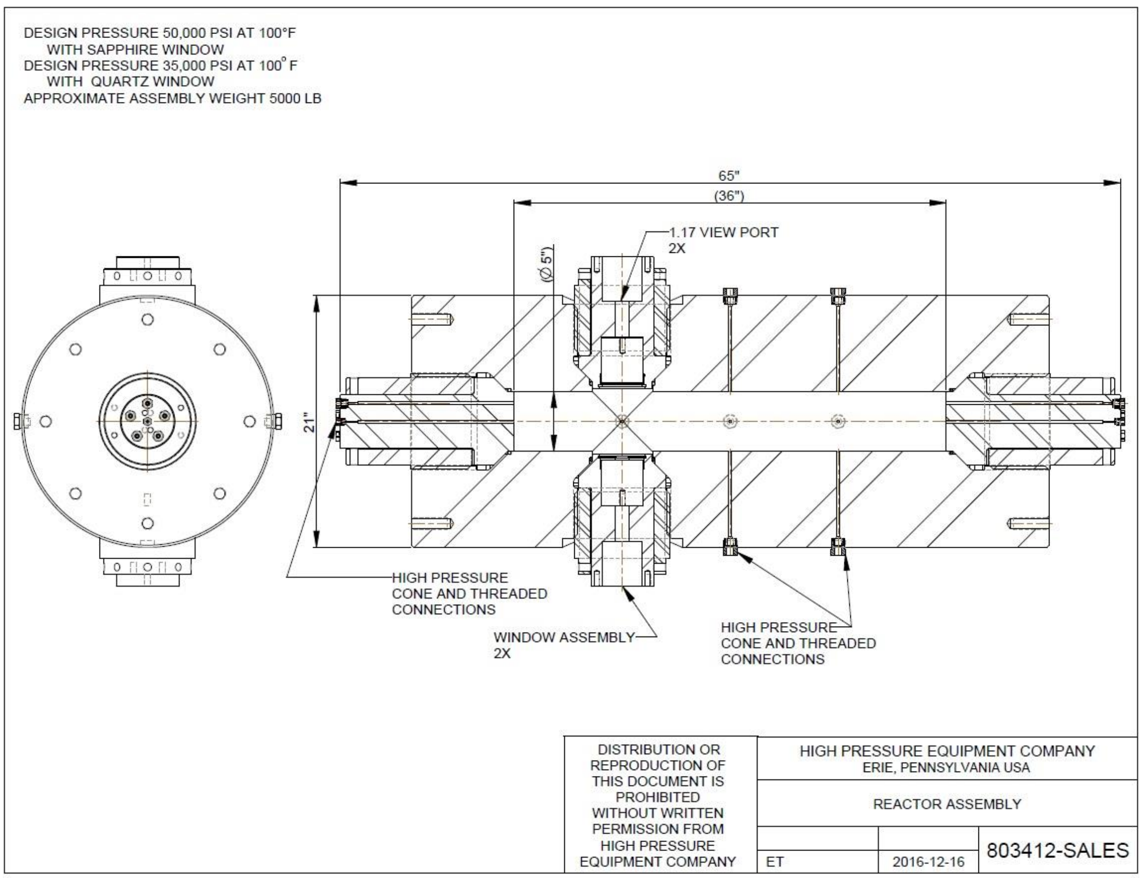

Figure 6. Design overview of chamber.

UHPOC Characteristics

The chamber is constructed from 4340 heat-treated steel, equipped with both optical viewing ports (1.17-in) for direct observation, and access ports installed with electronic feedthroughs to pass diagnostic signals through the chamber walls. For lower pressure testing, a temperature conditioning system can be coupled to the chamber to provide means to precondition reactants inside the chamber. A photograph of the chamber mounted on the test stand is shown in Figure 7.

The main body of the chamber has an overall length of approximately 53 inches with a 36-inch long internal free volume designated as the test section. Once both end-closures are installed, the overall length is nearly 66 inches. The inner diameter of chamber is five inches which yields a free volume of approximately twelve liters.

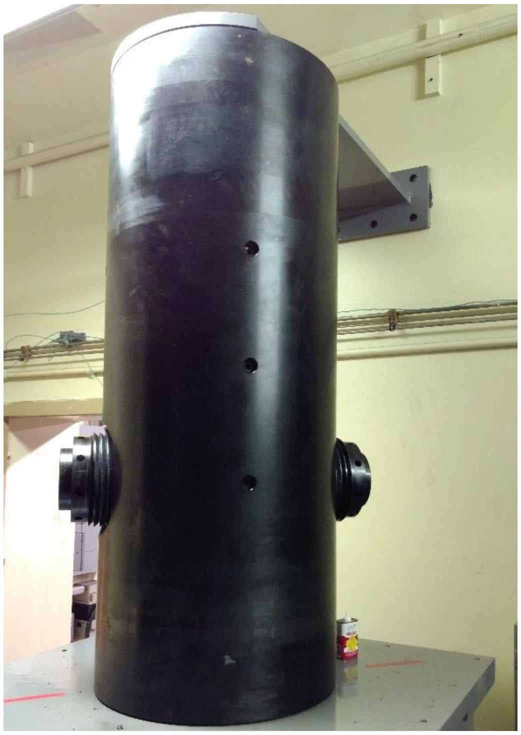

Figure 7. UHPOC featuring diametrically opposed window inserts; instrumentation access ports are visible in the foreground.

The chamber's large internal diameter provides enough room to house the multiple different kinds of experimental modules. Furthermore, the large free volume allows the reduction of severe pressure spikes during the combustion event. The outer diameter of the chamber is 21 inches which provides a chamber wall thickness of eight inches to allow windows to be installed and to maintain the internal pressure. The chamber ends employ identical end-closures. The window ports were specifically located favoring one side of the chamber and not located at the midpoint of the chamber to allow for short strand tests and the possibility of installing additional windows or access ports in the future. The chamber is equipped with ten instrumentation access ports at various locations along the chamber body and six access ports on each end closure. Any of the instrumentation ports can be used for gas inlet/exhaust points, fiber optics, instrumentation, thermocouples, pressure transducers, ignition, etc.

Current Configuration

The UHPOC has been currently configured for strand burning experimentation. Two access ports on the upper end-closure are equipped with a rupture disk and a pressure transducer. A manual pressure gage is fitted to the pressurant gas supply line immediately upstream of the chamber. This pressure gage is a redundant internal pressure measurement employed as safety precaution in the event of a failed pressure transducer signal. The remaining access ports can serve as pathways for bulk gas temperature measurements or other instrumentation. Circular optical access ports with a field of view of 1.17 inches provide the means to record the combustion event using high-speed cinematography. The window elements are sealed with O-rings located inside of the window holders and the window assembly mates to the chamber. A structural plate and test was designed and fabricated to mate the chamber to the test stand.

The main pressurant gas is provided to the chamber through one of the upper instrumentation ports on the main body. An inlet gas port can also be installed in the chamber's lower end-closure to provide a pathway for delivering inert gas (typically nitrogen or argon) during the experiment as well as regulating the chamber pressure. The exhaust port is located in one of the upper instrumentation ports on the main body to provide an exhaust exit for combustion product gases during the experiment. To regulate the chamber pressure during the experiment, remote metering valves will be used to control purge gases (argon or nitrogen) flowing through the inlet and exhaust ports.

Each of the end-closure units are equipped with six access ports; some of which are electronic feedthroughs installed to pass instrumentation signals or ignition current through the chamber wall. Two electrically isolated ignition posts are mounted in the bottom end closures. Electrical wiring passes through the interior of the end closure to provide means for ignition. The internal wiring is connected to external voltage supply circuitry to create a current through the nichrome wire igniter. Figure 8 shows the position of the strand burner and ignition posts when the lower end-closer is installed. The full strand burner mechanism is visible by removing one of the side windows.

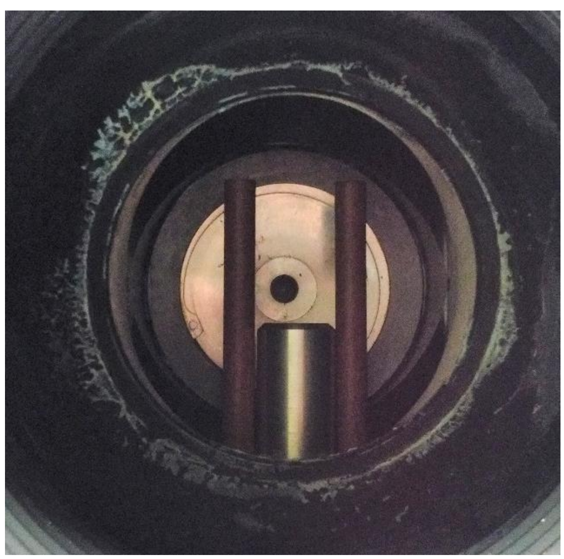

Figure 8. The strand burner insert with electrically isolated ignition posts visible through window opening

Installation and Auxiliary Instrumentation

To provide a mounting platform and support structure for the chamber, a test stand was manufactured from plain-carbon steel. The stand's upper plate is 1.5-in-thick with a 12-in circular cutout for access to the chamber's lower end-closure. This plate is supported by vertically mounted W36x135 I-beams which were attached to a slotted base for forklift access. A finite element simulation was performed on the stand to validate the hand calculations ran during design and to demonstrate suitability of the stand before installing the chamber. The results of this analysis, shown in Figure 9, predicted a maximum von Mises stress of approximately 4% of the yield stress of the material. The top plate showed stress concentrations on opposing sides of the opening, but due to the low stress levels, it was determined these concentrations would not result in a failure mode.

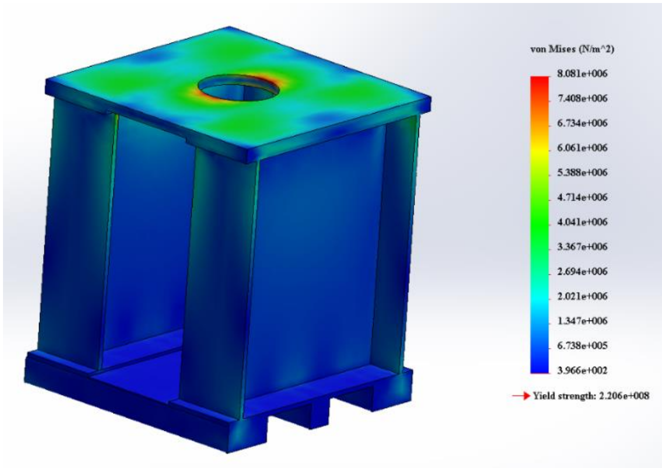

Figure 9. FEA analysis of test stand showed max stress ~4% of yield.

The final chamber had a weight of approximately 2300 kg. which necessitated the use of a crane and forklift to mount the chamber to the test stand. The mounting process was performed outside of the test facility and the chamber and stand assembly was moved into the building's test cell for final installation. After the assembly was placed in its final location, a support bracket connected to the chamber was bolted through the 18-in-thick reinforced concrete wall to anchor the chamber in the event of a sudden pressure release event. The progression of the chamber installation is shown in Figure 10.

Figure 10. (left) assembled mounting stand; (center) mounting of the chamber and stand; (right) chamber installed into test cell.

After the chamber was mounted within the test cell, the high-pressure plumbing for the chamber was installed. All high-pressure plumbing used for this project is rated at 414 MPa, giving a 114 MPa margin for the highest planned pressures of 300 MPa. A burst disc rated at 358.7 MPa was also fitted to the chamber as a safety mechanism. Gas flow to and from the chamber is controlled via a series of needle (HiP-60-11HF9) and pneumatic isolation valves (HiP-60-11HF9-MPO-NC and -NO), also rated at 414 MPa. The pneumatic valves are supplied the required 0.34 MPa of control gas via a low-pressure air compressor which is controlled by relay-actuated solenoids. Finally, exhaust gas from the chamber is filtered and then vented to atmosphere via low-pressure plumbing rated at 22 MPa. Figure 11 details the full PID for this setup, with images of the final plumbing and valve installations shown in Figure 12.

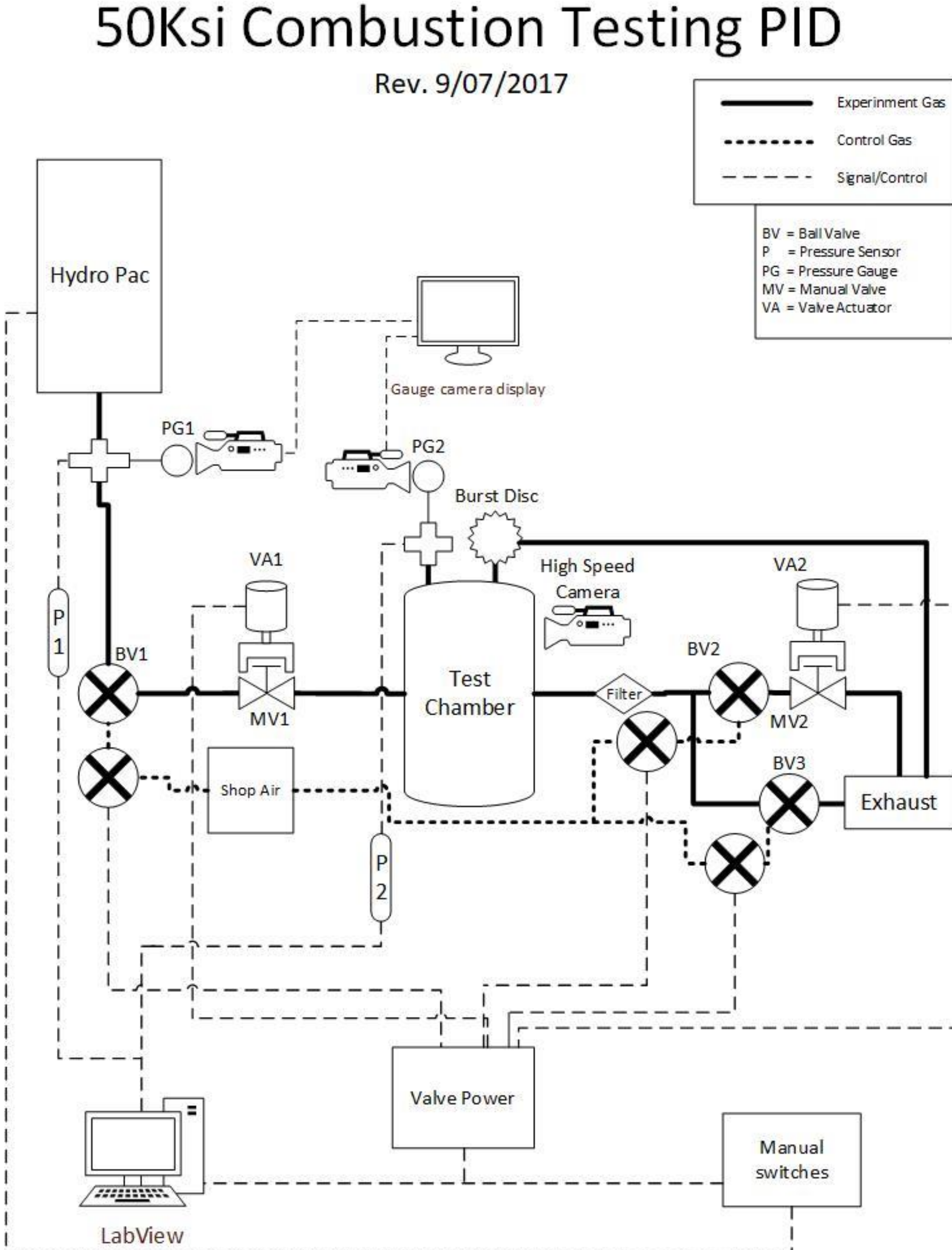

Figure 11. Project PID

To access the interior of the chamber, the lower end-closure of the chamber must be removed. This end-closure is a two-part sealing system (sealing plug and threaded collar) and weighs approximately 55 kg. The end-closure must be lifted precisely into the chamber under the tight constraints of the test stand interior. Moving this sealing system by hand would be prohibitive to most operators so a mechanism was created to facilitate movement. The mechanism, shown in Figure 13, lowers and lifts the plug and collar to the correct positions, as well as supports the test samples while they are being instrumented. This lifting mechanism is operated via an electric winch and guided by linear rails during vertical travel.

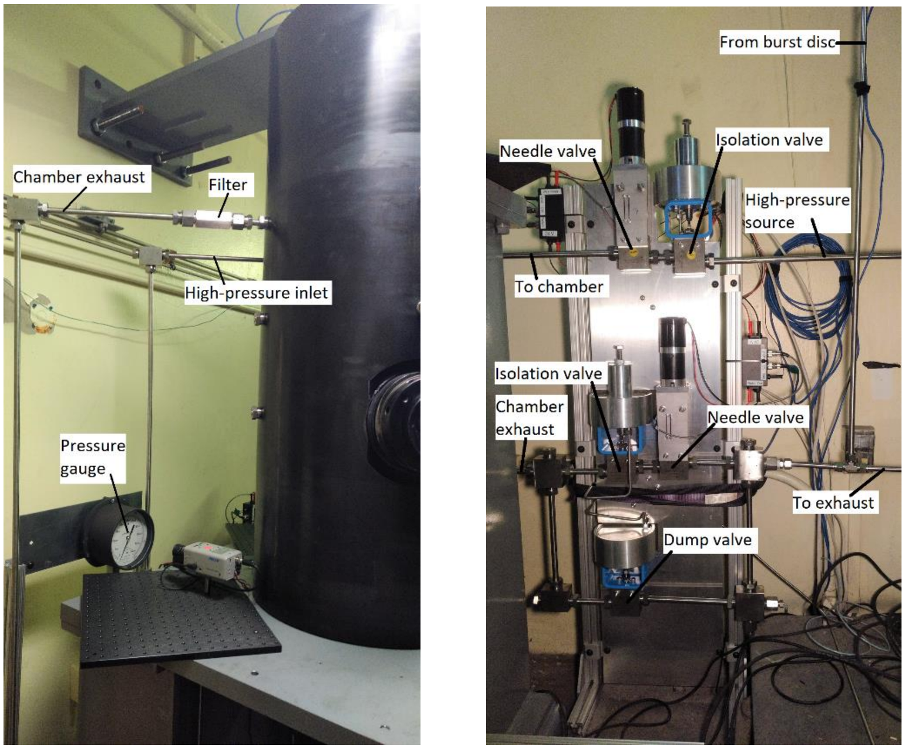

Figure 12. (left) chamber plumbing installation; (right) valve installation.

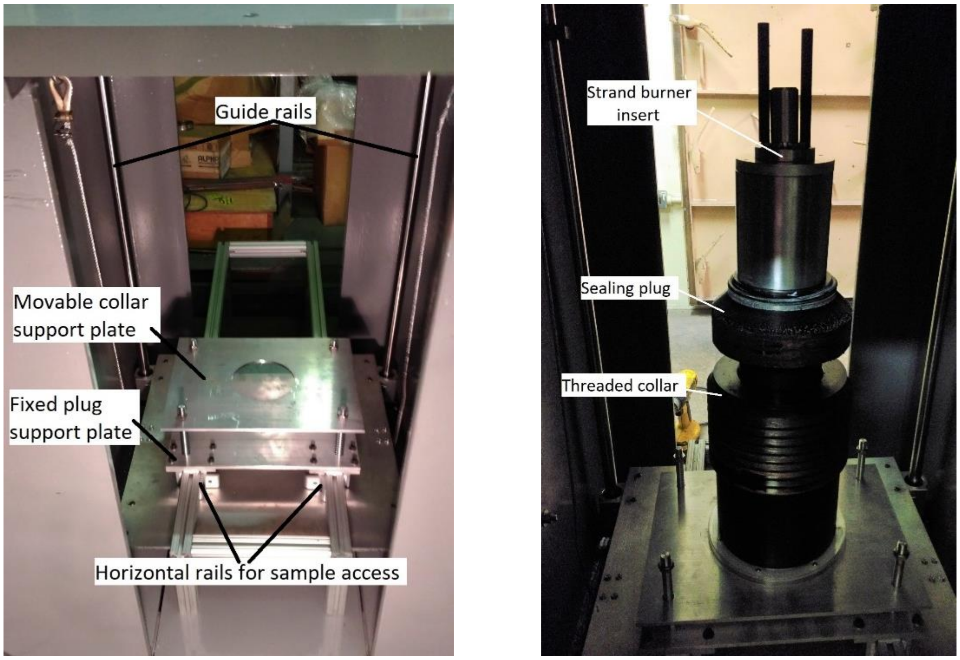

Figure 13. Lifting mechanism without (left) and with (right) sealing plug and strand burner

The rate of gas flow to and from the chamber is controlled by variable needle valves. These valves typically require hand operation to open and close which would put an operator at risk if an unforeseen event were to occur during an experiment. To eliminate the need for an operator to be present in the test cell, the valves were modified for remote operation via a DC motor.

Modular Inserts: Strand Burner

Chamber inserts provide modular diagnostics and/or experimental configurations. These inserts are also able to withstand the ultra-high pressures for which the chamber was designed (~ 345 MPa). For this DURIP, the strand burner insert was developed. This insert is shown in Figure 14 and is designed to accept a 1.5-in-long sample with a diameter of either 0.25 or 0.5 in. The sample holder is mounted on a perforated plate, also visible in Figure 14, which provides a path for an inert gas to flow over the sample during combustion to keep the viewing windows unobstructed. The sample holder is flanked by two electrically isolated posts which support the electrodes for nichrome-wire ignition. The insert assembly is raised off of the sealing plug by a hollow cylinder which both aligns the sample with the viewing windows and houses the electrical connections to the chamber exterior.

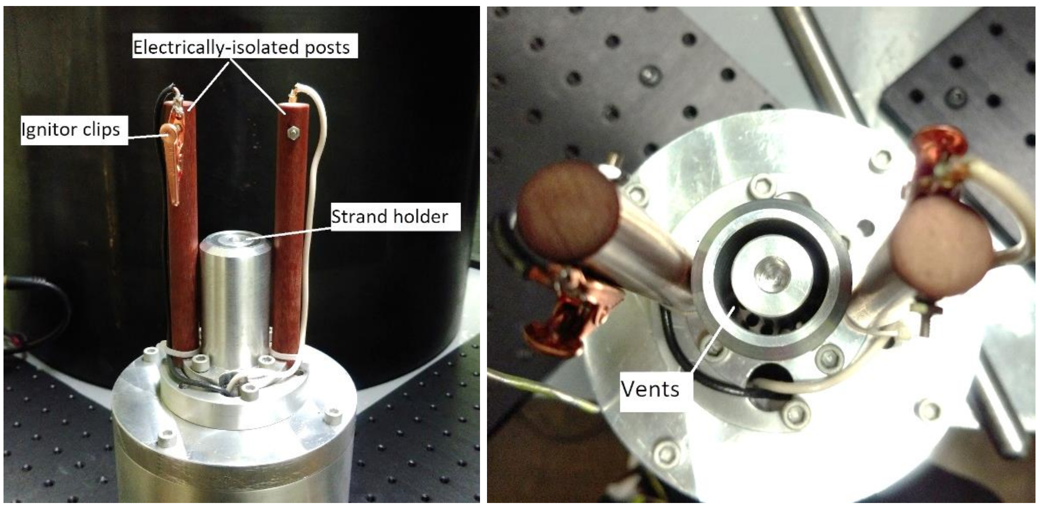

Figure 14. Strand burner insert with ¼-in strand insert installed

Control and Data Collection

The control system was built to emphasize redundancy and safety. Most features are controlled either directly through electrical hardware on the manual control panel or via a Measurement Computing USB-SSR24 relay controlled from LabVIEW. The user panels for both systems are shown in Figure 15 and are interconnected so that valve status, valve control, power status, pressure, and video are synchronized between the two. Because both systems are able to operate most of the systems independently, multiple control points must fail before control of the chamber is lost. A toggle switch on the hardware control panel selects which system is the primary. In the unlikely event that a complete loss of control occurs, the system fails to a safe state, venting any pressurized gas to atmosphere. While almost all of the control systems to the chamber are redundant, the remote operation of the needle valves is currently confined to just the hardware control panel. This decision was made to simplify the design of the control system for the initial setup. Future updates to the control systems could allow these valves to be redundantly operated.

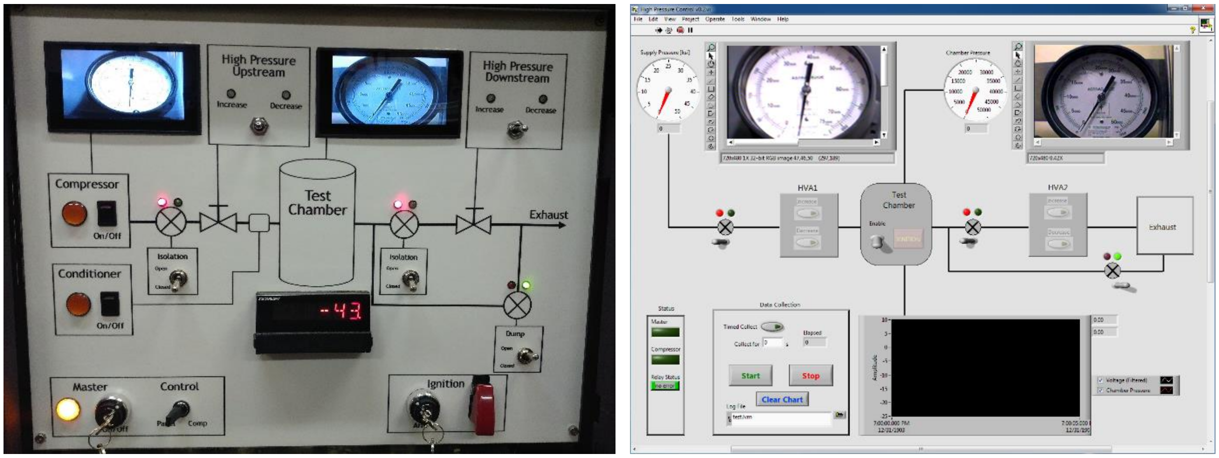

Figure 15. (left) hardware control panel; (right) software control panel.

Ignition of the propellant sample is accomplished by resistive heating of nichrome wire from a variable-voltage AC source. Operation of the ignition system is a two-step operation; both the hardware and software panels require two physical actions to energize the relay-controlled AC source. Depending on the type of experiment, ignition can also be achieved using CO_2 laser, propellant booster charge, or a combination of methods.

The control system is able to collect a wide variety of data for combustion experiments. A custom LabVIEW program monitors and records chamber pressure generated from an Omega PX91N1-50KS5T pressure sensor through a National Instruments USB-6361 data acquisition system. Visual burning-rate information is captured via a Phantom v310 high-speed camera, and a Nikon D40x digital-SLR captures high-resolution images of the experiment. Figure 16 shows the camera placements using a first-surface mirror setup. This data collection system is extensible, allowing collection of gas temperatures, sample subsurface temperatures, and supply pressure as needed by the experiment.

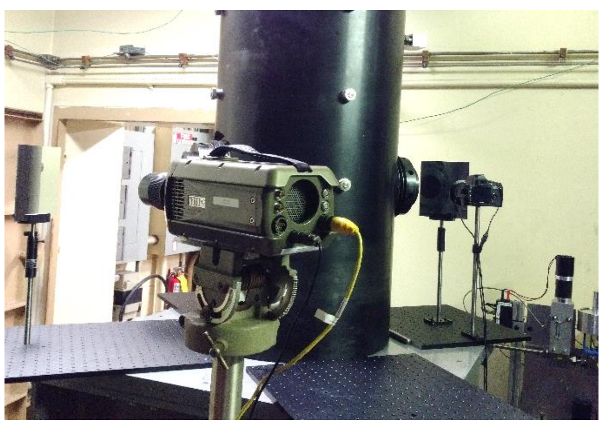
Figure 16. Remote image capture using high-speed and digital SLR cameras

Due to the extreme pressures involved with use of this chamber, the system was designed to have redundant pressure monitoring systems. In addition to using LabVIEW to monitor chamber pressure, pressure sensor data are also sent to a digital readout on the hardware control panel. To supplement the pressure sensor data, the analog pressure gauges on the chamber and high-pressure compressor are monitored with closed-circuit TV cameras. The video feeds from these cameras are sent to both the software and hardware control panels allowing viewing of both pressure sources regardless of the control method used. The camera monitoring the analog pressure gage is visible in the bottom of Figure 12 while the analog and digital displays are shown in Figure 15.

All subsystems described here have been fully verified. Experiments, data, and resulting analysis using the Universal High Pressure Optical Chamber will be reported under ARO grant W911NF1710149.

Summary

A new experimental chamber has been developed for studying combustion phenomena of advanced energetic materials. This Ultra-High Pressure Optical Chamber provides optical access for gathering visual data on combustion experiments from 0.03 to 345 Mpa. The chamber has been built and tested to the required pressures, and has been installed at the Kuo High Pressure Gas Lab at The Pennsylvania State University. Auxiliary systems needed to manipulate the chamber's components, gather data, and control the system have also been installed and validated.

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